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HIGH TEMPERATURE RESISTANT MATERIALS FOR MISSILE PROPULSION SYSTEMS

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UNITED STATES NAVAL ORDNANCE LABORATORY, WHITE OAK, MARYLAND

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HIGH TEMPERATURE RESISTANT MATERIALS FOR MISSILE PROPULSION SYSTEMS
Summary Report: 30 June 1966 - 30 June 1967

Prepared by:
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ABSTRACT: Oxy-acetylene burner studies were run on extra long specimens of phenolic asbestos using the alpha-rod technique, and it was shown that steady-state ablation was reached. Also tested were two densities of foamed silica. The high density (0.4 g/cc) foamed silica had the highest heat of ablation of any material tested to date except for phenolic filled zirconia foam. Calculation of alpha vs. temperature from experimental data was programmed for a digital computer, which also supplies a graph of alpha vs. temperature.

Verification of the digital computer program for predicting internal temperature profiles was carried out on the foamed silicas. The low density (0.2 g/cc) foam calculation was within 4% of the experimental time to 200°C, the best agreement to date. Extension of the technique to include rocket motor liners is in progress.

A detailed study of the NOL transient calorimeter was made. Generally, the existence of a linear temperature rise on the back face is sufficient evidence that errors are negligible.

Seven task groups in ASTM Committee E-21 are working to standardize methods and nomenclature for characterizing plasma arcs, for measuring temperatures of ablating bodies and for measuring char performance. Publication of several of these methods in the ASTM book of standards is imminent.

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HIGH TEMPERATURE RESISTANT MATERIALS FOR MISSILE PROPULSION SYSTEMS
Summary Report: 30 June 1966 - 30 June 1967

The high temperature materials program at the Naval Ordnance Laboratory is a continuing study of materials' behavior in high thermal environments. Information gathered in these studies is intended to serve as a guide in the design and selection of heat resistant materials and for the optimization of materials for specific ablation applications. This report is a statement of progress for the period 30 June 1966 - 30 June 1967. The work was conducted under the Naval Ordnance Systems Command Task ORD 033 305/092-1/F009-06-03, Problem No. 1, Missile Propulsion Materials: High Temperature Resistant Materials.

This report covers the work concerned with the performance of organic, char forming ablators and also ASTM ablation test standardization. The remainder of Problem No. 1 dealing with high viscosity thermal protection materials is covered in a separate report.

Many of the materials discussed in this report were obtained from commercial sources. Their evaluation by the Laboratory in no way implies Navy endorsement for their high temperature usage. Neither is this consideration of a material by the Navy to be used for promotion purposes. There is no implication intended that other materials might not have performed as well as those selected for these studies.

E. F. SCHREITER
Captain, USN
Commander

Albert Lightbody
ALBERT LIGHTBODY
By direction

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ILLUSTRATIONS

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INTRODUCTION

Program Objectives

Selection and sizing of rocket motor and nozzle materials are based largely on subscale and full-scale testing. Besides being expensive and time consuming, the data from one application are not easy to use for other applications without further testing.

The program at the Naval Ordnance Laboratory is aimed at developing laboratory methods of selecting and sizing rocket materials, thus minimizing the need for full-scale tests.

Approach

The general approach is to develop equations which describe the behavior of an ablator in terms of its effective thermal diffusivity, α^*, and ablation rate, u. Then α^* and u must be determined for the environmental conditions of each application. Laboratory facilities are used under as many different conditions as possible for these determinations, and on the basis of these tests α^* and u are correlated with the environmental parameters. The correlations are then extrapolated to subscale test conditions and, finally, to full-scale conditions. At each step in the procedure, experiments are used to verify and modify the correlations.

Progress During Fiscal Year 1967

During FY 1967, alpha-rod tests were run in the oxy-acetylene burner on extra long specimens of phenolic asbestos and on two densities of foamed silica. Calculations were performed to predict internal temperature profiles in the foamed silica specimens. Further improvements were also made in the area of data reduction for the alpha-rod test. Materials were ordered for rocket motor liner tests.

A detailed study of the NOL transient calorimeter was completed. This study included the effects of a thermal gradient in the slug, heat losses, variable thermal properties, variable heat flux, and surface effects.

Each aspect of work accomplished in FY 1967 is covered separately in the report.

OXY-ACETYLENE BURNER STUDIES

General

Three materials were tested during the year using the oxy-acetylene burner as the heat source. Specimens were held at a constant distance from the burner using the automatic feed device (ref. (a)). A transient slug calorimeter was used to measure the heat flux. The parameters measured in these tests were the ablation rate, internal temperature profile, total weight loss, and frontface temperature. The ablation rate was determined by using the best straight line through the relative position record of the automatic feed device. Internal temperature profiles were determined using chromel-alumel thermocouples located

0.953 cm (0.375 in.) from the front face of the specimen. Using these parameters the effective thermal diffusivity, α^*, was computed using the formula (ref. (b)):

$$\alpha^* = \frac{u^2(T - T_0)}{dT/dt}$$

where T is the temperature at time t, dT/dt is the slope of the temperature profile at temperature T, T_0 is the initial temperature, and u is the ablation rate. Also computed was the effective heat of ablation, h*, given by

$$h^* = \frac{\dot{q}\, At}{\Delta w}$$

where $\dot{q}$ is the heat flux, A is the area of the specimen exposed to the burner, t is the total test time, and Δw is the total weight loss.

Phenolic Asbestos

Extra long alpha rods of phenolic asbestos were tested at a nominal heat flux of 200 cal/cm^2 sec. These alpha rods provided three times the usual thickness to be ablated, thus giving a longer time to reach steady state. The data from these tests are compared with earlier tests (ref. (c)) on standard length alpha rods at 200 cal/cm^2 sec and 60 cal/cm^2 sec in Table I. Only the tests on the extra long alpha rods reached steady state. At 200 cal/cm^2 sec, the two lengths give similar results except for the effective thermal diffusivity. This difference arises because steady state was not reached on the standard length alpha rods. At 60 cal/cm^2 sec, the heat of ablation is about the same as at the higher heat flux, as expected. Compared with the higher heat flux, erosion rate and mass loss rate are lower by a factor of about 3.5, because the heat flux is lower by a factor of about 3.5. The effective thermal diffusivity is high because steady state was not reached.

Foamed Silica

Alpha-rod tests were also run on foamed silica specimens produced by Emerson and Cuming, Inc. These materials are interesting because of their low density and also their low dielectric constant. Two materials were used: a "high" density foam, 0.4 g/cc, and a "low" density foam, 0.2 g/cc. Photographs of these materials before and after testing at 60 cal/cm^2 sec are shown in Figures 1 and 2. Graphs of alpha vs. temperature are shown in Figures 3 and 4. The most important features of these graphs are the generally higher magnitude of α^* as compared to charring materials and the fact that α^* for the foamed silicas has a maximum in the same temperature region where charring materials exhibit a minimum. The other test parameters are given in Table II, along with values for phenolic asbestos and phenolic impregnated zirconia foam for comparison. It is interesting to note that while the densities of the two foamed silicas differ by a factor of two, their ablation rates differ by a factor of five and their effective diffusivities by a factor of eight. The heat of ablation of the high density foam is higher than that of any material tested to date except for the phenolic filled zirconia foam (ref. (c)). On a weight basis, the high density foam appears attractive for applications with low shear stress and

moderate heat flux. On a volume basis, the high density foam does not appear as attractive due to its relatively high ablation rate.

Improvements to Data Reduction

Near the end of last year, a digital computer program was written to calculate effective thermal diffusivity using alpha-rod test data (ref. (c)). During the past year, this program (originally set up for CEIR-BASIC language computer) was rewritten in the FORTRAN language for the IBM 7090 computer. Further instructions were added to take advantage of the CALCOMP automatic plotter. The present program now accepts millivolt (thermocouple) data from the alpha-rod test for direct conversion to alpha vs. temperature and temperature vs. time in the form of graphs and tables. Details of the digital program are included in Appendix A. A technique developed for converting millivolts to temperature is also described.

In its present form, the 7090 program is an efficient method of data reduction and, more important, we find it less demanding on project workers as was the case with the analog system.

Summary and Conclusions

Phenolic asbestos alpha rods only reach steady state in the oxy-acetylene burner when an extra long specimen is tested at a high heat flux. Attainment of steady state has been shown (ref. (c)) to be very important for the valid calculation of alpha, and this point should be carefully studied for each material.

High density foamed silica (0.4 g/cc) appears to be attractive, on a weight basis, for low shear stress, moderate heat-flux conditions. These results, plus the earlier good results with phenolic filled zirconia foam, point to foamed ceramics as a promising type of material that should be studied in more detail.

Digital data reduction was found to be the most convenient way to process the experimental results. Raw data, in the form of the millivolt-time output of the thermocouple (as well as the ablation rate and initial temperature), is programmed for the computer. The computer output is graphs of alpha vs. temperature and temperature vs. time, as well as tables of these quantities.

BACKFACE TEMPERATURE CALCULATIONS

General

One of the goals of this program is to predict the thickness of thermal protection material required for a given task. A method of accomplishing this has been devised (ref. (d)) which uses the effective thermal diffusivity, α^*, and the ablation rate, u, in the heat conduction equation with a moving boundary An IBM 7090 computer program is used to solve the differential equation numerically. While it may not be possible to obtain firm design data from this progra it should be possible to reduce appreciably the number of rocket motor test firings. To determine the accuracy of this method, calculated temperature profiles were compared with experimental profiles (ref. (c)).

Foamed Silica

During the past year, oxy-acetylene burner tests on foamed silicas at 64 cal/cm^2 sec were used to check the accuracy of the analytical method. Alpha vs. temperature data for a low density (0.2 g/cc) foamed silica was used first. A comparison of the calculated and experimental temperature profiles is shown in Figure 5. The calculated time of arrival of the 200°C isotherm was within 4% of the experimental time. This is the closest agreement obtained for any material tested to date.

Alpha vs. temperature data for a high density (0.4 g/cc) foamed silica was then used in the computer program. In this case, the calculated time of arrival of the 200°C isotherm was much longer than the experimental time, as shown in Figure 6. This large difference arises because the high density foamed silica specimen did not reach steady state. Very similar results were obtained in previous tests (ref. (c)) on phenolic asbestos, which also failed to reach steady state.

Rocket Motor Liner Studies

A more stringent check of the computer program would be the calculation of temperature profiles of cylindrical specimens in the exhaust of a subscale rocket motor. Preparations are being made for blast tube test series both in the NOL hydrogen-oxygen rocket motor and the larger liquid propellant engine at the Naval Weapons Center, China Lake, California (formerly NOTS).

A blast tube fixture for the NOL rocket motor was successfully proof tested with a silica filled epoxy liner in the tube. Chamber pressure was maintained throughout the test and there were no leaks. Only minor modifications of the thermocouple location will be needed for future testing.

A series of reinforced phenolic blast tube specimens have been ordered for rocket motor testing. The materials ordered for the NOL rocket motor are graphite phenolic, Pluton phenolic, Refrasil phenolic, and nylon phenolic. The materials ordered for the Naval Weapons Center's liquid propellant motor are graphite phenolic and Pluton phenolic. In addition, specimens made from promising NOL epoxy-novolak resin systems reinforced with Pluton have been fabricated for both motors.

Summary and Conclusions

The calculated temperature profile for low density foamed silica agreed with the experimental profile within 4%, the best agreement to date. High density foamed silica did not reach steady state and so showed poor agreement.

Having checked the analytical method for several materials tested in the oxy-acetylene burner, the next step is to check the method for materials tested in subscale rocket motors. Preparations for such tests were made both for the NOL hydrogen-oxygen rocket motor and the Naval Weapons Center's liquid propellant engine.

CALORIMETRY

General

Heat flux is the most important parameter describing an ablation test environment. A transient slug calorimeter has been in use at NOL for about seven years. This calorimeter was used in the A.S.T.M. standardization of the NOL panel test (ref. (e)). In addition, the calorimeter is used in wind tunnels to characterize aerodynamic flow conditions (ref. (f)). In view of the widespread use of this instrument and others like it, a detailed study of its characteristics was made. A formal report on the calorimeter will be published separately and only the highlights will be presented here.

Ideal Calorimeter

An ideal calorimeter consists of a lossless slug with constant thermal properties and constant initial temperature. Assuming a constant uniform heat flux suddenly applied to the front face, the temperature profile in the slug can be found analytically. The backface temperature rises linearly after a transient time of about 0.7 sec, in our case. On the linear portion, the calorimeter equation is valid.

$$\dot{q} = \rho c L \dot{T}$$

$\dot{q}$ = heat flux

ρ = slug density

c = slug specific heat

L = slug length

$\dot{T}$ = time derivative of backface temperature

This equation holds regardless of a temperature gradient in the slug, contrary to the frequent assumption that there must be no gradient for valid results.

Real Calorimeter

A real calorimeter does not meet the conditions required for an ideal calorimeter exactly. Heat losses tend to make the temperature profile droop below the predicted linear rise. Losses limit the present calorimeter to a minimum of about 20 cal/cm^2 sec. Also, the heat flux into the slug, from a constant environment, decreases as the slug heats up. This variation of heat flux is usually not as important as the losses.

Another source of error is the variation of slug thermal properties with temperature. Numerical calculation on a digital computer showed that this produces a 5% error at 200 cal/cm^2 sec, unless the average values of the thermal properties are used.

Surface catalytic effects can also be important in convective heating. If a significant fraction of the environment gas is dissociated, the measured heat flux will be low unless the calorimeter surface catalyzes recombination. A copper-faced calorimeter, such as ours, can read 20% lower than a silver-faced calorimeter in non-equilibrium flow (ref. (g)).

Summary and Conclusions

A transient calorimeter can be used in both convective and radiant heat sources. Major sources of error were examined with the following results: the thermal gradient in the slug does not cause any error, heat losses are the limiting factor at low heat flux, the variation of thermal properties with temperature can be accounted for by using an average value, the variation of heat flux was negligible in these tests, and catalytic recombination is potentially important in convective heating but must usually be considered on a per case basis.

Generally, the existence of a linear temperature rise on the back face is sufficient evidence that the errors are negligible. However, this does not account for catalytic effects and a relatively small correction for variable thermal properties. The calorimeter is expected to be accurate within ± 5% when used routinely in the oxy-acetylene burner.

ASTM ABLATION COMMITTEE WORK

General

During the past year, the ablation group in the American Society for Testing and Materials (ASTM) Committee E-21 on Space Simulation continued its efforts to standardize methods for characterizing plasma arcs, methods for measuring the temperatures of ablating bodies, methods for measuring char performance, and definitions and nomenclature pertaining to ablation. The above work areas are divided among seven groups each with a separate chairman. The overall (section) chairman is Mr. F. J. Koubek of the Naval Ordnance Laboratory. Individual Task Groups and their respective chairmen are listed below:

Section 3 on Ablation

Chairman - F. J. Koubek, U. S. Naval Ordnance Laboratory
Secretary - R. H. Reid, U. S. Polymeric Inc.

Task Group No.	Task Group Title	Chairman
1	Flame Testing	Inactive
2	Definitions and Nomenclature	M. A. Schwartz, IIT Research Inst.
3A	Enthalpy of Plasma Arcs-Energy Balance	H. Hoercher, AVCO Corporation
3B	Enthalpy of Plasma Arcs-Probe Techniques	J. Grey, Greyrad Corporation
4	Heat Flux and Calorimetry	J. Todd, Consultant
5	Rocket Materials Testing	W. Andreport, Air Force Rocket Propulsion Laboratory

Section 3 on Ablation (continued)

Task Group No.	Task Group Title	Chairman
6	Internal Temperature Measurements	S. Grindle, Space General Corp.
7	Surface Temperature Measurements	R. Bierman, General Electric Co.

Section 3 met twice during fiscal year 1967: on 24-25 October 1966 at San Francisco, California and on 3 May 1967 at Toronto, Canada. The following is a summary of what this group accomplished during the fiscal year toward its objectives, which are to develop tests, recommended practices and techniques for measuring the properties of ablative materials.

Methods for Measuring Plasma Arc Performance

Three task groups are working to develop standards and recommended practices for measuring the heat flux and enthalpy of plasma arcs.

Task Group 3A has prepared a proposed method for measuring total enthalpy by energy balance. This method was letter balloted jointly in section-subcommittee with favorable results. Of the 98 returns, 72 were affirmative (13 with comments) and 26 were "not voting." There were no negative votes. In response to the suggestions put forth by those voting affirmatively "with comments," a number of minor editorial changes were made to the proposed method. At the Toronto meeting in May 1967 the revised method was approved for full Committee E-21 letter ballot. If this ballot is favorable, the method will be sent to ASTM headquarters for final review and publication.

In essence, the method is intended as a measure of the total or stagnation enthalpy of a direct current, plasma arc gas stream by means of an overall system energy balance. The determination of total enthalpy is based upon the following measurements: (1) energy input to the plasma arc, (2) energy losses to the plasma arc hardware and cooling water, and (3) gas mass flow. The gas enthalpy is determined by dividing the gas mass flow into net power input to the plasma arc (power to the plasma arc minus the energy losses). The test method is mainly concerned with accurate measurement of the above items to get a reliable total enthalpy determination.

Task Group 3B is working toward the preparation of proposed methods or recommended practices for measuring enthalpy using probe techniques. Enthalpy probes provide a measurement of enthalpy at specific points in the plasma stream, whereas the total enthalpy (Task Group 3A) provides an overall average for the stream. Since enthalpy probes are relatively new to the field, Task Group 3B is surveying the "status-of-the-art" to determine which, if any, techniques are suitable for standardization.

At our spring meeting, Dr. Grey presented a report on his latest survey, and the following conclusions were drawn:

1. The tare-measurement calorimetric probe has become a standard, reliable and accurate device for subsonic or stagnation point measurements at high densities, where its limited sensitivity is not a disadvantage. However, it is not considered a sufficiently general purpose instrument to form the basis of a "standard practice" recommendation.

2. For supersonic and hypersonic environments, the existing blunt-nosed non-tare measurement probe appears suitable for stagnation point measurements and for measurements requiring high-sensitivity capability but has not had sufficient use to qualify as a standard device.

3. The development of a sharp-lipped (shock-swallowing) supersonic/hypersonic enthalpy probe, also suitable for mass flux measurements, has begun and some encouraging experimental data have been recorded. Problems in heat transfer, size reduction, sharp-tip fabrication, and inlet fluid mechanics, however, have yet to be solved. The sharp-lipped probe appears to provide the general purpose capability but is still far from the stage of evaluation and development at which a recommendation could be made.

4. The fast response probe has not yet been evaluated sufficiently. In particular, definition of calibration requirements and exposure to high heat transfer environments are still lacking. This design shows sufficient promise, however, to remain as a potential candidate for standardization, pending further development.

5. All other types of enthalpy probes do not appear to qualify for standardization, although reasonably good data have been obtained with the modified split flow design. Special purpose devices such as the cooled electrostatic probe will always find specific applications suited to their unique features.

Since neither the tare measurement probe nor the sharp-lipped probe can be considered as "standard techniques" at this time for the reasons given in 1 and 3 above (and no other probe techniques qualify at present), Dr. Grey recommended that additional time be allowed for development, and that the situation be reviewed again in six months, at which time it is possible that a clear choice can be made.

Task Group 4 is working toward the preparation of standard procedures for measuring heat flux by means of transient and steady-state calorimeters. A proposed recommended practice for measuring plasma arc heat flux using a transient (slug) calorimeter has not yet been released from the group.

Methods for Measuring the Temperature of Ablating Bodies

Task Group 6 has prepared a proposed recommended practice for measuring internal temperature histories of ablating bodies in a high temperature gas stream. Specifically, the proposal deals with the proper use of thermocouples. Some of the problem areas treated are: wire diameter, thermocouple junction area and its orientation to heat flow, junction placement accuracy in relation to the heated surface, electrical shorting by conductive char, and the relation of thermocouple assembly effective conductivity to ablator conductivity.

This recommended practice was letter balloted jointly in section and subcommittee with favorable results. Of the 94 returns, 71 were affirmative (3 with comments), 2 were negative and 21 were "not voting." In response to the suggestions put forth by those voting negative or "with comments", a number of minor editorial changes were made to the recommended practice. At the Toronto meeting in May 1967, the revised document was approved for full Committee E-21 letter ballot. If this ballot is favorable, the method will be sent to ASTM headquarters for final review and publication.

Task Group 7 is maintaining liaison with Sub-Committee VI (on Radiation Thermometers) of ASTM Committee E-21 and is presently advising them on the type of standard test method needed for measuring surface ablation temperature. Section 3 members will be polled to learn of their preferences and operating experiences in this area.

Methods for Measuring Char Performance

Task Group 5 is working toward the preparation of methods and or recommended practices for measuring char and erosion depth. To aid in the preparation of standards, this group has established definitions for: pyrolysis depth, char thickness, surface recession, char-virgin interface, surface recession rate, and char recession rate. During the next fiscal year the group will work on recommended practices for char thickness, pyrolysis depth, and surface recession depth and thermocouple instrumentation in ablative rocket motor liners.

Definitions and Nomenclature

Task Group 2 completed two section letter ballots of the term "ablation" during the fiscal year. The initial ballot was of the term as it appears in NASA's "Glossary of Terms in High Temperature Protection Technology." The principal criticisms of this definition were of its lengthiness, adequacy, and accuracy. A shorter version was felt to be more complete in its simplicity. A second letter ballot of a shortened version was favorable and resulted in only one word (thermal) being added to the definition, which now reads: "Ablation--A self regulating heat and mass transfer process in which incident thermal energy is expended by sacrificial loss of material." In its present form, this definition is simple, concise and accurate and it will probably not require further modification as new uses arise for this form of thermal protection. At the Toronto meeting in May 1967, the revised document was approved for full Committee E-21 letter ballot and subsequent publication.

Balloting of definitions for "effective heat of ablation," "heat of ablation" and "ablation efficiency" as they appear in the NASA glossary are slated for fiscal year 1968.

Summary and Conclusions

A proposed standard method for measuring plasma arc enthalpy and a recommended practice for installing thermocouples in ablative test specimens have been balloted favorably at sub-committee level. Full E-21 committee balloting and subsequent publication by ASTM is expected during FY 1968. Other methods for measuring specimen performance in terms of char and erosion depth are in preparation. The

areas of enthalpy probe techniques and surface temperature and heat flux measurement techniques are under continued surveillance for future standardization. Balloting of a proposed method for the measurement of heat flux using a transient slug calorimeter is expected in FY 1968. Final balloting of the term "ablation" and subsequent publication by ASTM is anticipated during FY 1968. Three additional terms are also slated for letter balloting.

The above progress represents the continued efforts of a volunteer group working in a somewhat difficult area. The complexity of the ablation process and the large variety of service environments and simulation equipment make it difficult to arrive at specific standardized tests of use to more than a few groups. However, there are a number of subprocedures common to many tests that need to be standardized for an orderly dissemination of performance data for maximum effective use by ablation technologists.

It is essential that the Navy and other DOD activities and NASA continue to encourage and support the activities of the ASTM Committee on Ablation. In this way, there will be a continued production of standardized procedures badly needed in the ablation field.

RECOMMENDATIONS AND FUTURE PLANS

Experimental verification of the digital computer program for predicting internal temperature profiles should be extended to two more of the typical materials already tested in the oxy-acetylene burner. In order to round out and complete these studies, fast ablating, highly insulating materials should be studied. The material types selected are rubber and cork based materials.

With the background and experience obtained in the parametric studies and experimental verification, initial rocket motor studies should begin. Internal temperature histories and ablation rates will be obtained in blast tube studies in the NOL hydrogen-oxygen rocket motor. Further tests will be done in a liquid propellant motor at the Naval Weapons Center. The experimental data will be compared with predicted temperature histories based on alpha-rod test measurements, and, if needed, refinements will be made to the program to improve its accuracy.

The above rocket motor studies should give some indication of the adequacy of current alpha-rod data to predict ablative materials requirements and whether data are needed over a broader range of test conditions not available in the oxy-acetylene burner. If this is the case, access to a more versatile test device, such as a large gas plasma arc heater (ref. (b)), will be necessary to continue the program.

TABLE I

ALPHA-ROD STUDIES OF PHENOLIC-ASBESTOS
IN THE OXY-ACETYLENE BURNER*

	Tested at a Nominal Heat Flux of 200 cal/cm^2 sec		Tested at a Nominal Heat Flux of 60 cal/cm^2 sec
	Standard Length Alpha Rod	Extra Long Alpha Rod	Standard Length Alpha Rod
Ablation Rate 10^{-3} cm/sec	14.1	14.2	3.52
Mass Loss Rate 10^{-2} g/sec	6.52	7.46	2.03
Heat of Ablation kcal/g	8.8	7.7	8.0
Effective Thermal Diffusivity at 400°C 10^{-4} cm^2/sec	8.5	4.0	8.5

* Oxy-acetylene burner design and gas flow conditions are the same as those set forth in ASTM E285-65T.

TABLE II

SUMMARY OF ALPHA-ROD, OXY-ACETYLENE*
TEST RESULTS ON FOAMED SILICA

Specimen	Density g/cc	Ablation Rate 10^{-3} cm/sec	Mass Loss Rate 10^{-2} g/sec	Heat of Ablation kcal/g
High density foamed silica	0.4	7.62	0.89	18.
Low density foamed silica	0.2	40.4	2.34	6.8
Phenolic-Asbestos	1.7	3.52	2.03	8.0
Phenolic impregnated ZrO_2 foam	1.6	1.45	0.28	58.

* Oxy-acetylene burner design and gas flow conditions are the same as those set forth in ASTM E285-65T.

APPENDIX A

DIGITAL METHOD OF DATA REDUCTION

The first step in the calculation of the effective thermal diffusivity is to convert the millivolt output of the thermocouple to degrees centigrade. Since this is a common operation, the details may be of interest to others. It is desirable to find an analytic relation for temperature as a function of millivolts, not only because of the convenience of a formula but also because it is easily used in digital computers. A fit was made to tabular data, reference (A-a), for chromel-alumel thermocouples with a 0°C reference junction. The method of least squares was used to fit a polynomial to the table. The highest temperature of interest to us is about 1200°C, which corresponds to 54.2 mv. In using a large number, such as 54.2, in the method of least squares, errors result from raising this large number to high powers. The calculation is therefore more accurate if a reduced variable, R, is introduced that varies from minus one to plus one. Negative values are further desirable since some cancellations take place. Then,

$$R = \frac{MV - 27.1}{27.1}$$

A polynomial of sixth degree was found to be adequate for the temperature interval from 20°C to 1200°C,

$$T = c_o + c_1 R + c_2 R^2 + c_3 R^3 + c_4 R^4 + c_5 R^5 + c_6 R^6$$

$c_o = 651.609$

$c_1 = 639.985$

$c_2 = 28.4603$

$c_3 = 46.9802$

$c_4 = -23.6123$

$c_5 = -12.0537$

$c_6 = 18.7761$

Once the temperature profile is known, the effective thermal diffusivity can be calculated from the formula

$$\alpha^* = \frac{u^2(T - T_o)}{dT/dt}$$

Since the temperature profile is roughly exponential, it is convenient to use the natural logarithm of the temperature. Then

$$\alpha^* = u^2/\frac{d}{dt} \ln(T - T_o)$$

The method of least squares was used to evaluate the derivative and provide some smoothing of the data at the same time. Five equally spaced, nearby points are chosen, a second degree polynomial is fitted to these points by the method of least squares, and the derivative is evaluated at the mid-point. The final result is (ref. (A-b))

$$\frac{dY}{dt} = \frac{-2Y_{-2} - Y_{-1} + Y_1 + 2Y_2}{10h}$$

where $Y = \ln (T - T_o)$, h = time interval between points, and the subscript on Y refers to the five points, labeled -2, -1, o, 1, and 2. The derivative above is valid at point o.

This analysis was first programmed in BASIC computer language. Five parameters must be read into the program:

U = ablation rate, in./sec

B = initial temperature, °C

H = time interval between points, sec

N = number of points

L = total test time, sec

In addition, N values of temperature, in millivolts, must be read in. The output of this program is a table of time vs. temperature vs. alpha. The BASIC program is given below:

```
100 REM CALCULATIØN ØF EFFECTIVE THERMAL DIFFUSIVITY

110 DIM C(100), M(100), K(100), A(100), T(100), R(100), D(100)

120 READ U, B, H, N, L

130 LET F = 2.54*U

140 FØR I = 1 TØ N

150 READ M(I)

160 LET R(I) = (M(I) - 27.1)/27.1

170 LET D(I) = 651.609+639.985*R(I)↑2+28.4603*R(I)↑2+46.9802*R(I)↑3

180 LET C(I) = D(I)-23.6123*R(I)↑4-12.0537*R(I)↑5+18.7761*R(I)↑6

190 LET K(I) = LØG(C(I)-B)

200 NEXT I
```

```
210 FØR I = 1 TØ N
220 LET A(I+2) = (10*H*F↑2)/(-2*K(I)-K(I+1)+K(I+3)+2*K(I+4))
230 NEXT I
240 PRINT "ABLATIØN RATE ="F"CM/SEC ("U"IN/SEC)"
250 PRINT "TEST TIME ="L"SEC    AMBIENT TEMPERATURE ="B"DEG C"
260 PRINT "TIME", "TEMPERATURE", "ALPHA"
270 PRINT "0", C(1)
280 PRINT H, C(2)
290 FØR I = 3 TØ N-2
300 PRINT (I-1)*H, C(I), A(I)
310 NEXT I
320 PRINT (N-2)*H, C(N-1)
330 PRINT (N-1)*H, C(N)
340 END
```

The same analysis was also programmed in FORTRAN for the IBM 7090 computer. One important advantage of the 7090 computer is that graphs can be generated and plotted automatically by the CALCOMP plotter, ref. (A-c). The following parameters must be read in:

UENG = ablation rate, in./sec

TO = initial temperature, °C

TESTIM = total test time, sec

TMAX = desired time range on graph, sec

H = time interval between points, sec

N = number of points

In addition, N values of temperature, in millivolts, must be read in. In addition to the tabular output as before, two graphs are plotted: temperature vs. time and alpha vs. temperature. The temperature scale on the graphs goes from 0 to 1200°C in both cases, the time scale goes from 0 to TMAX, and the alpha scale goes from 0 to 40 x 10^{-4} cm^2/sec. The FORTRAN program is given below.

```
C     CALCULATION OF EFFECTIVE THERMAL DIFFUSIVITY
      DIMENSION XMV(100),R(100),TEMP(100),XL(100),ALPHA(100),
     1 TIME(100),HDG(30)
  100 READ (5,110) J,(HDG(I),I=1,2)
  110 FORMAT (I1,2A6)
      IF (J.NE.1) STOP
      WRITE (6,110) J,(HDG(I),I=1,2)
      READ (5,120) UENG,TO,TESTIM,TMAX,H,N
  120 FORMAT (5E10.5,I5)
      READ (5,130) (XMV(I),I=1,N)
  130 FORMAT (7E10.5)
      UMET=2.54*UENG
      DO 140 I=1,N
      R(I)=(XMV(I)-27.1)/27.1
      Z=R(I)
      TEMP(I)=(((((18.7761*Z-12.0537)*Z-23.6123)*Z+46.9802)*Z+28.4603
     1 )*Z+639.985)*Z+651.609
  140 XL(I)=ALOG(TEMP(I)-TO)
      DO 150 I=5,N
  150 ALPHA(I-2)=(10.*H*UMET**2)/(-2.*XL(I-4)-XL(I-3)+XL(I-1)+2.*XL(I))
      WRITE (6,160) UMET,UENG,TESTIM,TO,H,N
  160 FORMAT (25H0ABLATION RATE, CM/SEC = F7.5,10X,24HABLATION RATE, IN/
     1SEC = F7.5,10X,19HTEST TIME, SEC    = F6.1/25H0AMBIENT TEMP., DEG C
     2  = F6.1,11X,24HTIME INTERVAL, SEC    = F6.1,11X,19HNUMBER OF POIN
     3TS = I3/9H0    TIME,5X,9H    TEMP.,5X,8H   ALPHA/)
      TIME(1)=0.
      DO 170 I=2,N
  170 TIME(I)=TIME(I-1)+H
      DO 210 I=1,N
      IF(I.GE.3.AND.I.LE.N-2) GO TO 190
      WRITE (6,180) TIME(I),TEMP(I)
  180 FORMAT (1H0F8.1,5X,F8.1)
      GO TO 210
  190 WRITE (6,200) TIME(I),TEMP(I),ALPHA(I)
  200 FORMAT (1H0F8.1,5X,F8.1,5X,4PF8.1)
  210 CONTINUE
      CALL CALCM1 (N,TIME,TEMP,0,0.,TMAX,0.,1200.,10.,6.,HDG   ,-12,9HTIM
     1E, SEC,-9,18HTEMPERATURE, DEG C,18,0.,18)
      CALL CALCM1 (N-4,TEMP(3),ALPHA(3),0,0.,1200.,0.,.004,6.,8.,HDG,-12
     1   ,18HTEMPERATURE, DEG C,-18,40HEFFECTIVE THERMAL DIFFUSIVITY, CM
     2SQ/SEC,40,0.,18)
      CALL CALCM1 (0,0.)
      GO TO 100
      END
```

TESTED FRONT VIEW

TESTED SIDE VIEW

UNTESTED SIDE VIEW

FIG. 1 PHOTOGRAPHS OF HIGH DENSITY FOAMED SILICA

UNTESTED SIDE VIEW

TESTED SIDE VIEW

TESTED FRONT VIEW

FIG. 2 PHOTOGRAPHS OF LOW DENSITY FOAMED SILICA

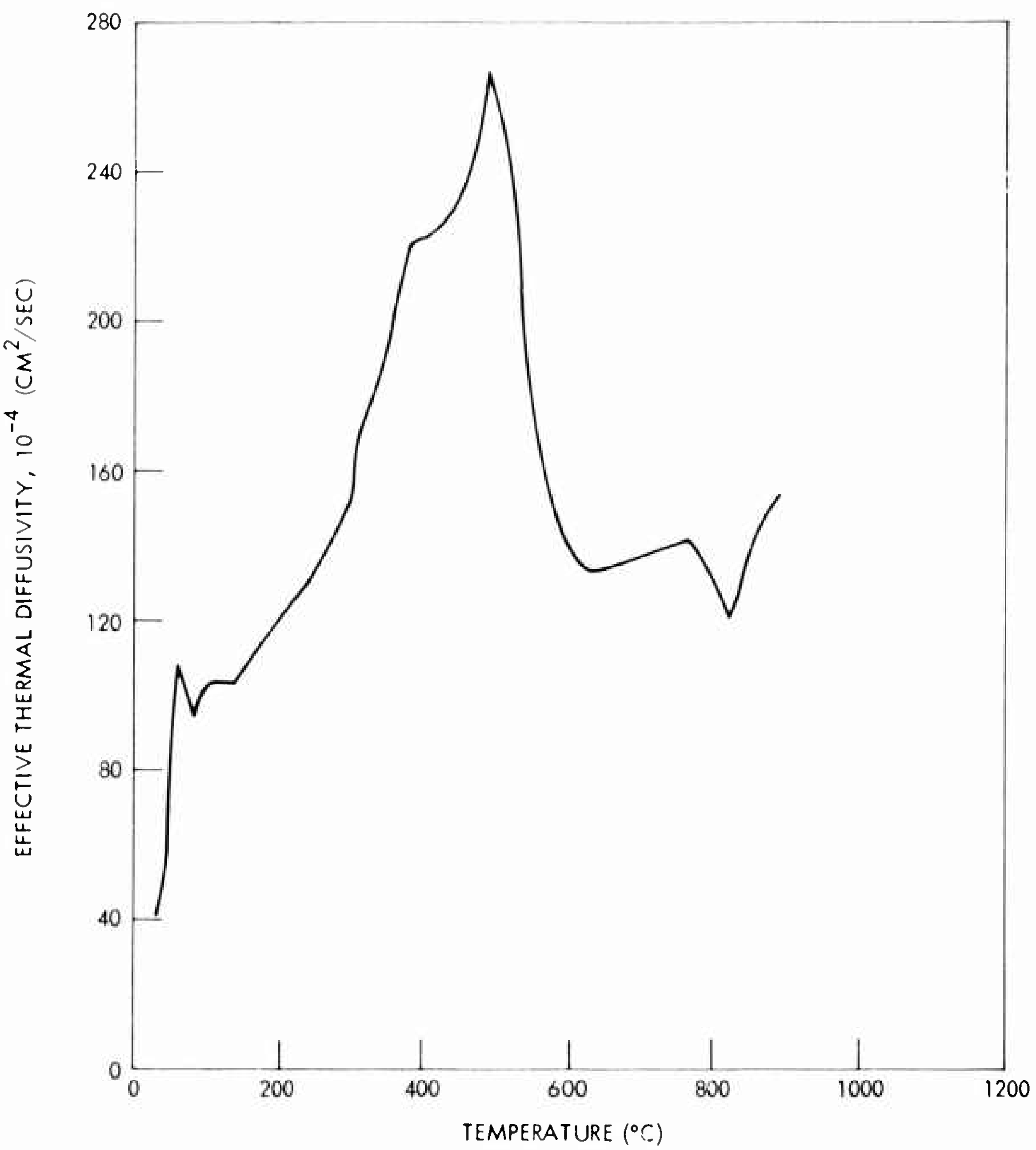

FIG. 3 EFFECTIVE THERMAL DIFFUSIVITY VS TEMPERATURE FOR LOW DENSITY FOAMED SILICA

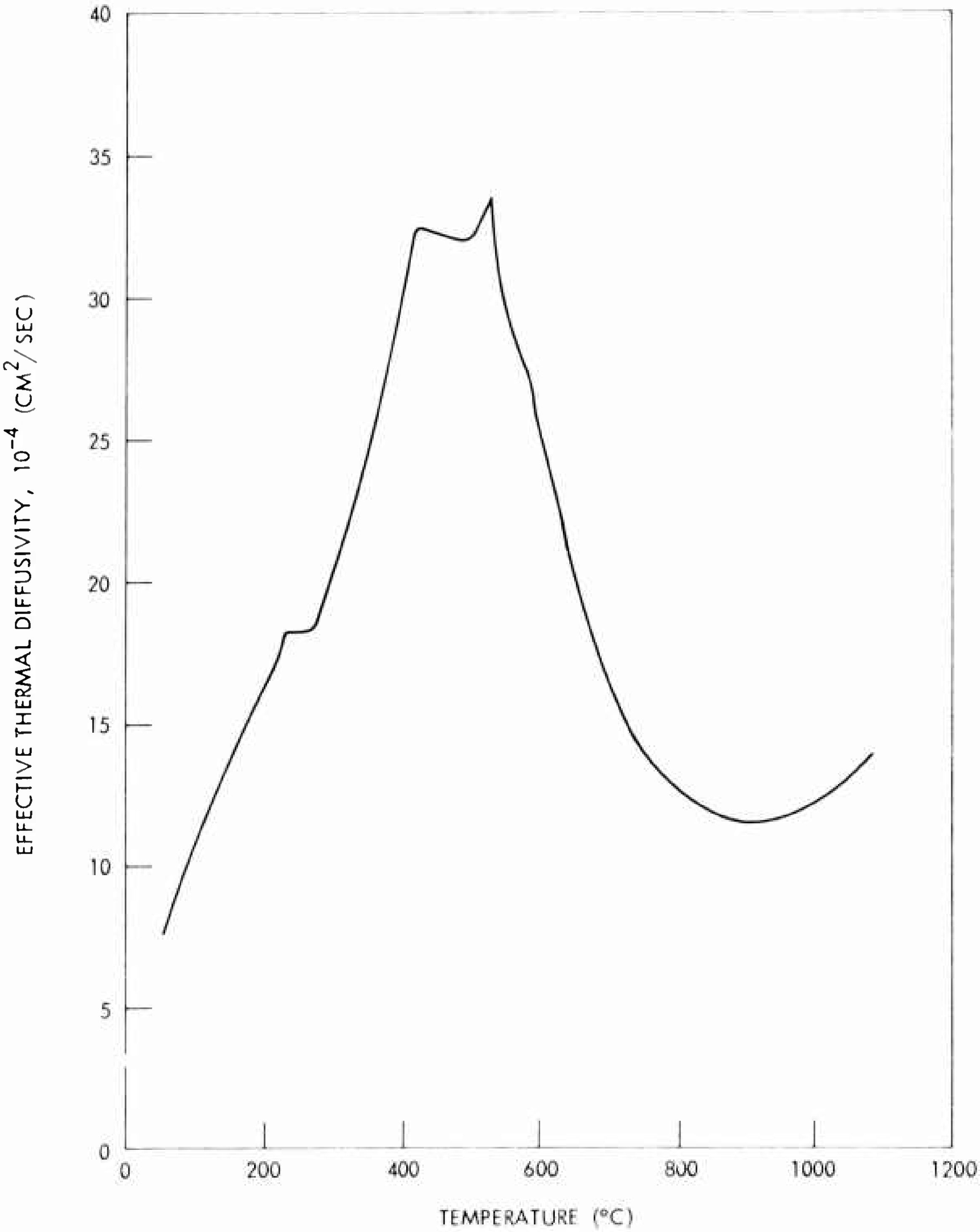

FIG. 4 EFFECTIVE THERMAL DIFFUSIVITY VS TEMPERATURE FOR HIGH DENSITY FOAMED SILICA

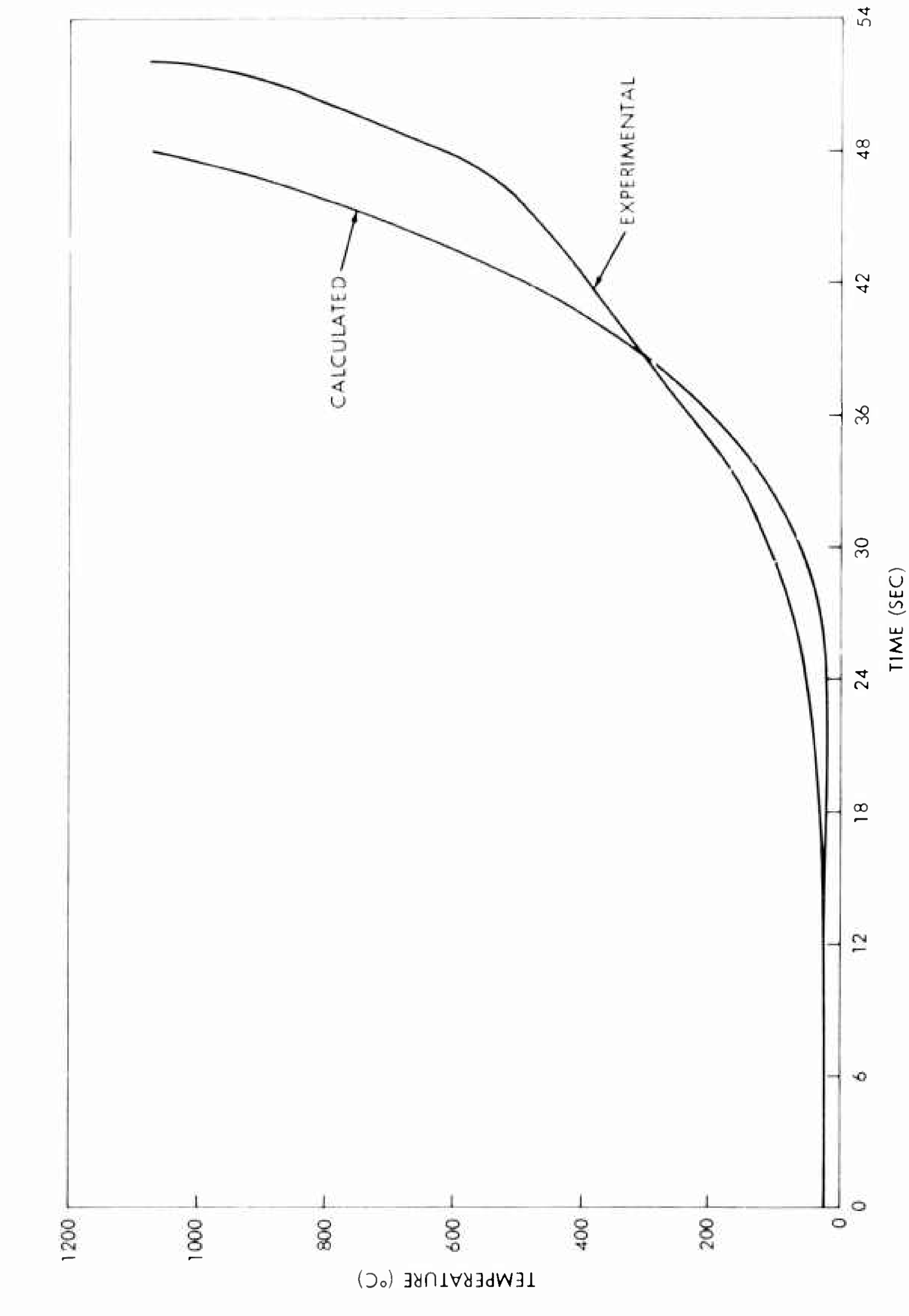

FIG. 5 EXPERIMENTAL AND CALCULATED TEMPERATURE PROFILES FOR LOW DENSITY FOAMED SILICA

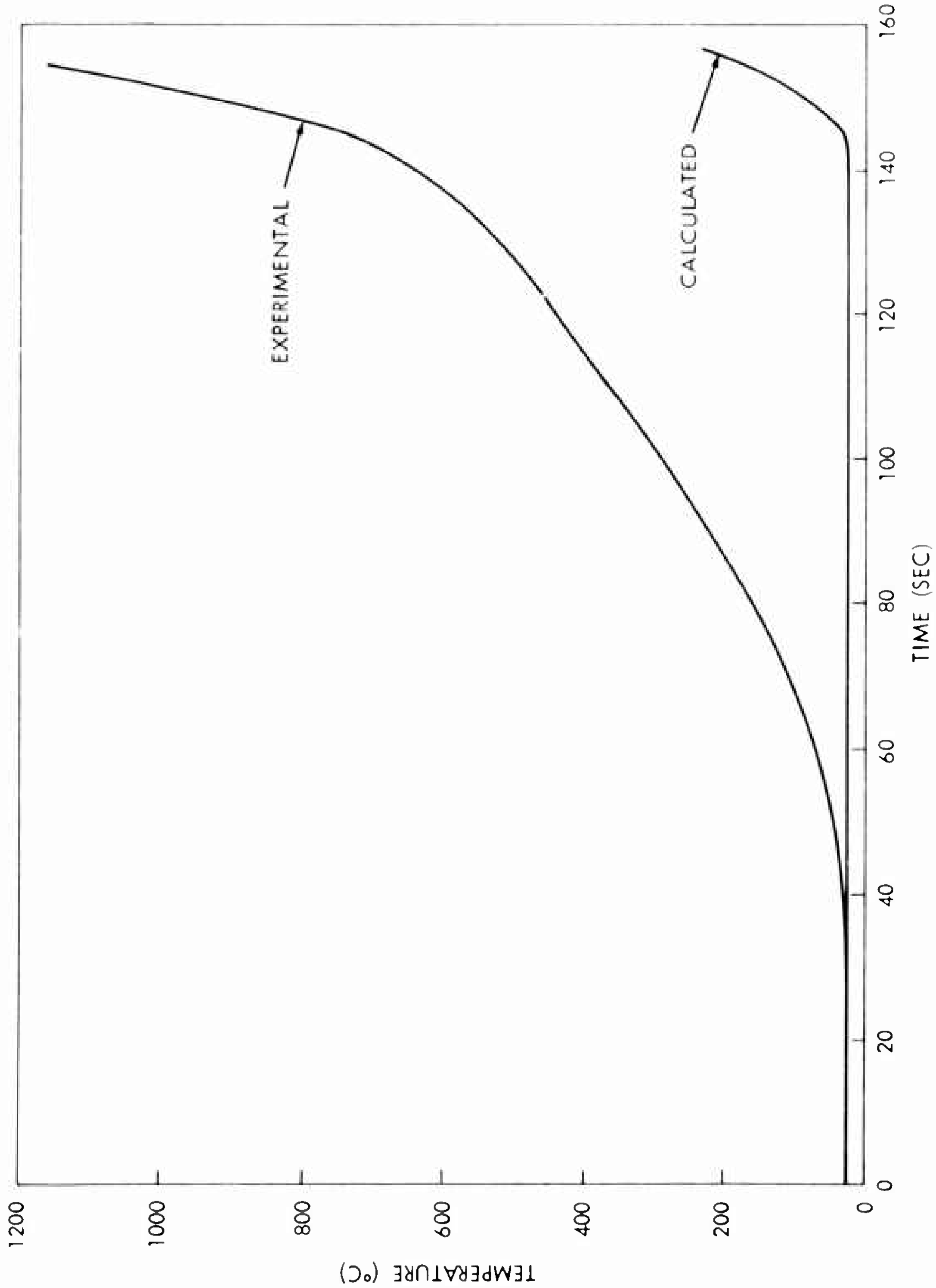

FIG. 6 EXPERIMENTAL AND CALCULATED TEMPERATURE PROFILES FOR HIGH DENSITY FOAMED SILICA

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)

U. S. Naval Ordnance Laboratory
White Oak
Silver Spring, Maryland 20910

2a. REPORT SECURITY CLASSIFICATION

Unclassified

2b. GROUP

3. REPORT TITLE

High Temperature Resistant Materials for Missile Propulsion Systems, Summary Report: 30 June 1966 - 30 June 1967

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Annual, 30 June 1966 - 30 June 1967

5. AUTHOR(S) (First name, middle initial, last name)

Bruce Hartmann
F. J. Koubek

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
9 October 1967	26	10

8a. CONTRACT OR GRANT NO.

ORD 033 305/092-1/~~F009-06-03~~

b. PROJECT NO.

Problem No. 1

c.

d.

9a. ORIGINATOR'S REPORT NUMBER(S)

NOLTR 67-146

9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

10. DISTRIBUTION STATEMENT

Distribution of this document is unlimited.

11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY

Naval Ordnance Systems Command
U. S. Navy Department
Washington, D. C. 20360

13. ABSTRACT

Steady state ablation was reached in oxy-acetylene burner tests on extra long specimens of phenolic asbestos. Also tested and analyzed were two densities of foamed silica. Calculation of alpha vs. temperature from experimental data was programmed for a digital computer.

A detailed study of the NOL transient calorimeter was made. Generally, the existence of a linear temperature rise on the backface is sufficient evidence that errors are negligible.

Seven task groups in ASTM Committee E-21 are working to standardize methods and nomenclature for ablation testing. Publication of several of these methods in the ASTM book of standards is imminent.

DD FORM 1 NOV 65 1473 (PAGE 1)

S/N 0101-807-6801

UNCLASSIFIED

Security Classification

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Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Materials, High Temperature						
Rocket Motors, Materials						
Ablative Materials						
Heat Shield Materials						
Plastics, Heat Resistant						
Temperature Prediction, Ablative Materials						

DD FORM 1 NOV 65 1473 (BACK)

(PAGE 2)

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Security Classification